

Name I.C. LEWIS / T. PIRRO
Notebook Number 195-130
Subject CHEMISTRY OF RAW MATERIALS, NEW BINDERS, + IMPREGNANTS
Dates From _____ To _____

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Subject 2nd VI of 195-129-97 #3-2-B w/ silicon (II) oxide, colloidal dispersion (c/c comp BP) 1
 Cross-Reference (if any) ---

Purpose:

Ref. 195-129-99

Materials:

- 1) c/c composite 195-129-97 #3-2-B. (c/c composite via BP process from the 2nd block of the 3rd Lawrenceburg trial. 0.25" long K-223-SE pitch fibers. Reilly 155 g. tub. Load ratio = 75 lbs w/b sulfur. VIed in w/ SiO₂ colloidal dispersion \Rightarrow hot vac. dried. Wt(w) = 861.58g, Vol(w) = 571.40 cc, Den(w) = 1.508 g/cc, Wt(pvd-1) = 903.59g, Current Den = 1.581 g/cc
- 2) Impregnant: silicon (II) oxide, 30% in H₂O, colloidal dispersion. (Alfa-Aesar) Lot # A04K09. 0.01 μ m particles, in liquid. SA = 320 m²/g, Density = 1.20, Load Used: 7/26/01 vis(w) = 7.7 cps at 82.1 °F, SG(w) = 1.216 at 82.1 °F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16 * Processed w/ 195-129-97 #3-2-A

Pump-Down Data: (7/27-30/01) - Process w/ 195-129-97 #3-2-A

DATE	TIME	PRESS (mm)	Comments
7/27	8:40	16	Load block from hot vac. oven (~166 °C, 0.3 mm)
"	8:50	"	Begin pump-down
"	10:00	22	Sample is dry \Rightarrow VI today
"	10:45	14	Charge traps w/ dry ice - acetone
"	11:45	9	LDR
"	13:00	9	Begin VI

Impregnation Data: (7/27-30/01) - LDR w/ traps charged.

LDR: Initial = 9 m Torr Vis(w) = 8.0 cps at 76.2 °F Prop. Time = 13:00 (9 m Torr)
 5 min = 21 " SG(w) = 1.220 at 76.2 °F Unload Time = 8:00 (7/30/01)
 10 min = 30 " Held at atmospheric pressure for
 15 min = 41 " ~ 67 hrs.

Comments:

~450 cc of impregnant in the 500 cc cylindrical funnel.

Post-Impregnation Data: (7/30/01)

Wt (PVI-2) = 1011.33g \Rightarrow Wt Pickup = 107.74g \Rightarrow Wt% Pickup = (11.92), Vol% Pickup = (15.43)

Performed and Recorded by: [Signature]

Directed by: [Signature]

Read and Understood by:

Date

Date

Date

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Subject ^{2nd VI of 195-129-97 #3-2-B w/ silicon (IV) oxide colloidal dispersion (ck comp BP)}
 Cross-Reference (if any)

Drying Data: (7/30-31/01) - Over set "29" (50%). Argon purge at 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:20	112	Atm	Load over; # to bot, rear of tray. Over set "29" (50%). Argon purge 5.0 SCFH (AIR)
- (3)	11:20	114	"	Unload over. Set over at "38" (50%). Weigh block hot.
	-	-	-	Wt = 981.44g \Rightarrow Wt Pickup = 77.85g \Rightarrow Wt/Lb Pickup = <u>8.62</u> (Yd = <u>72.3%</u>)
*	11:30	108 (N)	Atm	Load over; # to top, front of tray. Over set "38" (50%). Argon purge 5.0 SCFH (AIR)
(2)	13:30	130	"	Unload over. Set over at "46" (50%). Weigh block hot. Install new gasketing
	-	-	-	Wt = 942.14g \Rightarrow Wt Pickup = 38.57g \Rightarrow Wt/Lb Pickup = <u>4.27</u> (Yd = <u>35.8%</u>)
*	14:00	135 (A)	741.3	Load over; # to bottom, rear of tray. Vac. pump on. Argon purge off.
- (18)	8:00	166	0.3	Vac. pump off. Pressurize w/ argon. Set over at "48" (50%)
	-	-	-	Wt = 929.57g \Rightarrow Wt Pickup = 25.98g \Rightarrow Wt/Lb Pickup = <u>2.88</u> (Yd = <u>24.1%</u>)

Comments:

1) After 3 hrs, $\sim 113^\circ\text{C}$, atm. pressure;

Set over at "38" (50%). Patches of white residue, more than 195-129-100 #3-2-A, on exterior faces (i.e. SiO_2). Rotate 180° ; # to ~~bottom~~ top, front of tray

2) After 2 hrs, $\sim 130^\circ\text{C}$, atm. pressure;

Set over at "46" (50%). Wipe condensed H_2O from oven door. Remove old gasketing and install new.

3) After 18 hrs, $\sim 165^\circ\text{C}$, vacuum;

Set over at "48" (50%). Very small amount of white solid (SiO_2) beneath this block. Powdery, white residue on exterior faces, more than 195-129-100 #3-2-A.

Comments:

As w/ the #3-2-A sample the impregnant's % in situ weight yield is lower than expected (i.e. $\sim 31\%$) \Rightarrow run-out, or the previous SiO_2 from the 1st VI was dissolved during the 2nd VI.

Cumulative Wt Pickup = 67.99g \Rightarrow Cumulative % Wt Pickup = 7.89
 Previous " " " = 42.01g \Rightarrow Previous % Wt Pickup = 4.88

Label 195-~~129~~-2 #3-2-B

Performed and Recorded by

Directed by: J. C. Lee

Read and Understood by:

Date

Date

Date

Subject 195-129-98 #4-13-A1 w/195-129-53 (sub by vel 3422/Rinal) (ch. comp 33) 3
Cross-Reference (if any)

Purpose:

To determine the composition, properties, and physical test (thermal bleed). Prepared one is in L. cell.

Materials:

- 1) The composite: 195-129-98 #4-13-A1 (ch. composite via BP process) From the 13th block of the 4th Lawrenceburg trail. 0.25" long, pitch Fibers + Reilly 105 Fibers. Lead ratio = 25/25 w/o solder. Empirical end to ~3000°C., $\text{Wt.} = 277.03\text{g}$, $\text{Vol.} = 162.42\text{cc}$, $\text{Dens.} = 1.70\text{g/cc}$. Note: This material had 1 PT and reburied prior to graphitization.
- 2) Impregnant: 195-129-53 (sub by vel. GP-1432/Furinal). Prep. 5/14/01, last used: 5/17/01. Second reimpregnated. $\text{Vis.} = 17.4\text{cps at } 74.5^\circ\text{F}$, $\text{S.G.} = 1.188 \text{ at } 74.5^\circ\text{F}$, $\text{Hardness} = 34.4$ ($T = 0.26, n = 3$)

Apparatus:

Q5, 195-129-10

Process:

Ref. 195-129-15312 * Processed w/195-129-98 #4-13-A1

Temp. Lower Dates: (7/30/01)

DATE	TIME	PROG (min)	Comments
7/30	7:50	16	Lead from warehouse at atm. pressure.
"	8:00	"	
"	9:00	30	⇒ samples are dry
"	11:05	22	Charge traps w/ electrolyte - 900 to 1000
"	11:35	12	LPD
"	13:15	"	Begin XT

Impregnation Dates: (7/30 + 31/01) - Lead w/ traps charged.

LPD: Initial = 12 mTorr $\text{Vis.} = 20.1\text{cps at } 70.8^\circ\text{F}$
 $\text{5 min} = 25$ " $\text{S.G.} = 1.174/\text{cf at } 70.8^\circ\text{F}$
 $\text{10 min} = 34$ "
 $\text{15 min} = 44$ "

Prep Time = 13:15 (12 mTorr)
 Unleaded Time = 8:15 (7/31/01)
 Held at atmospheric pressure
 for ~ 17 hrs

Comments:

400 ml of impregnant in 500 ml cylindrical funnel.

Post Impregnation Dates: (7/31/01)

Graph: Ref. 195-130-03

Wt. (PVE-1) = 317.11g ⇒ Wt. Pickup = 40.06g ⇒ Wt. to Pickup = 14.47, w/o pickup (26.61)

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

4

Subject

Cross-Reference (if any)

VS₂ Core (202°C) of 195-129-98#4-13-A1 w/ 195-129-53

(Clickmap BP)

Curing Data: (7/31 + 8/1/01) - Cured w/ VI. d 195-129-98#4-13-B1. Samples placed on s.s. screen over small Al pan to determine amount of run-out.

Al Pan + Screen wt (g) = 191.07g

	TIME	OVEN SET	OVEN TEMP	Comments
*	8:40	48	164	Load oven. Purge w/ argon at 50 SCFH (AIR)
-(2)	10:40	"	167	Unload to desiccator. Set oven at "82" (50°C). Cool composite \Rightarrow weight
	-	-	-	Wt = 292.87g \Rightarrow Wt. Pickup = 15.84g \Rightarrow Wt/o Pickup = <u>5.72</u> (Yld = <u>39.5%</u>)
*	13:30	82	246	Load oven; rotate 180°, reverse position. Purge w/ argon at 50 SCFH (AIR)
-(2)	15:30	"	256	Unload to desiccator. Cool overnight + weigh next morning. Oven set "29" (50°C)
	-	-	-	Wt = 291.55g \Rightarrow Wt. Pickup = 14.52g \Rightarrow Wt/o Pickup = <u>5.24</u> (Yld = <u>36.2%</u>)

Comments:

1) After 2 hrs, ~166°C, atm. pressure;

Run-out evident on s.s. screen and in Al pan. Pan + Screen + Run-out = 195.62g \Rightarrow

Cured run-out = 4.55g (ie, ~~Total oven residue = 20.39g \Rightarrow Yld = 50.27%~~)

Set oven at "82" (50°C), Rotate sample 180° and reverse position w/ the "B1" sample.

2) After 2 hrs, ~251°C, atm. pressure;

Set oven to "29" (50°C). Transfer samples to desiccator. Cool overnight and weigh 8/1/01.

Post Curing Data: (8/1/01)

Pan + Screen + Cured Run-out_(g) = 195.71g \Rightarrow Cured Run-out = 4.64g

Wt/o = 291.55g \Rightarrow Wt. Pickup = 14.52g \Rightarrow Wt/o Pickup = 5.24 (Corrected Yld = 36.2%)

\Rightarrow Impregnant Yield (including run-out) = $[(4.64 + 14.52) / 40.08] \times 100 = 47.8%$

Label 195-130-04 * Taken by Dr. Huang w/o label 8/1/01. for machining

\Rightarrow Impregnant Yield (including run-out) Ref. 195-130-06, had to include both wt. pickups for each sample

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject VI + Cure (~250°C) of 195-129-98 #4-13-B1 w/195-129-53
Cross-Reference (if any)

(c/c COMP BP). 5

Purpose:

Ref. 195-130-03

Materials:

- c/c Composite: 195-129-98 #4-13-B1 (c/c Composite via BP process. From the 13th block of the 4th Lawrenceburg trial. 0.25" K-223SE Fibers. Reilly 155p. 7ch. Load ratio = 75/25 w/o sulfur. Graphitized to ~3000°C. Note: This material had no PI.
 $Wt_{(c)} = 255.92g$, $Vol_{(c)} = 165.987cc$, $Dens_{(c)} = 1.542 g/cc$
- Impregnant: 195-129-53 (w/50 by vol. GP-5432 / Furfural). Prep. 5/14+15/01. Last Use: 5/17/01. Stored refrigerated. $Wt_{(c)} = 17.4g$ at $74.5^\circ F$, $S.G._{(c)} = 1.188$ at $74.5^\circ F$, % Mod MCC = 34.4 ($\sigma = 0.26$, $n = 3$).

Apparatus:

Ref 195-120-15

Procedure:

Ref. 195-120-15+16 * Proceeded w/195-129-98 #4-13-A1

Pump-down Data:

DATE	TIME	PRESS (mm)	Comments
7/30	7:50	16	Load from warm oven at atm. pressure
"	8:00	"	
"	9:00	30	⇒ samples are dry.
"	11:00	22	Charge traps w/dry ice-acetone.
"	11:35	12	LDR
"	13:15	12	Begin VI

Impregnation Date: (7/30+31/01) - LDR w/traps charged

LDR Initial = 12 atorr $V_{(c)} = 20.42ps$ at $70.8^\circ F$ Drip Time = 13:15 (12 atorr)
 $5min = 25$ " $S.G._{(c)} = 1.194$ at $70.8^\circ F$ Unload Time = 8:15 (7/31/01)
 $10min = 34$ " Held at atmospheric pressure
 $15min = 44$ " for ~19 hrs

Comments:

400 ml of impregnant in 500 ml cylindrical funnel.

Post Impregnation Data: (7/31/01)

$Wt_{(PVI-1)} = 308.96g \Rightarrow Wt_{Pickup} = 53.04g \Rightarrow Wt_{to Pickup} = 20.73$, $Vol_{to Pickup} = 26.77$

Performed and Recorded by: 

Directed by: J (low)

Read and Understood by:

Date

Date

Date

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Subject V_E and Cure (vac) of 195-129-98 #4-13-B1 w/ 195-129-53. (c/c comp. A)
 Cross-Reference (if any)

Curing Data: (7/31 + 8/1/01) - Cured w/ Vied 195-129-98 #4-13-A1. Samples placed on SS. screen over small Al pan to determine amount of run-out.

Al Pan + Screen Wt = 191.07g

	TIME	OVEN SET	OVEN TEMP	Comments
*	8:40	48	164	Load oven. Purge w/ argon at 5.0 SCFH (AIR)
-(2)	10:40	4	167	Unload to desiccator, set over at "82" (vac). Cool overnight \rightarrow weigh.
	-	-	-	Wt = 277.36g \Rightarrow Wt Pickup = 21.44g \Rightarrow Wt/o Pickup = <u>8.38</u> (Yd = <u>40.4%</u>)
*	13:30	82	246	Load oven. Rotate 180°, reverse position. Purge w/ argon at 5.0 SCFH (AIR)
-(2)	15:30	"	256	Unload to desiccator. Cool overnight + weigh next morning. Over set "29" (vac)
	-	-	-	Wt = 275.68g \Rightarrow Wt Pickup = 19.76g \Rightarrow Wt/o Pickup = <u>7.72</u> (Yd = <u>37.3%</u>)

Comments:

1) After 2 hrs, $\sim 166^\circ\text{C}$, atm. pressure;

Run-out evident on SS screen and in Al pan. Pan + screen + Run-out = 195.62g \Rightarrow
 Cured run-out = 4.55g

Set over at "82" (vac). Rotate sample 180° and reverse position w/ the "A1" sample.

2) After 2 hrs, $\sim 245^\circ\text{C}$, atm. pressure;

Set over to "29" (vac). Transfer samples to desiccator. Cool overnight and weigh 8/1/01.

Post Curing Data: (8/1/01)

Pan + Screen + Cured Run-out: Ref. 195-130-04

Wt (CF) = 275.68g \Rightarrow Wt Pickup = 19.76g \Rightarrow Wt/o Pickup = 7.72 (Inv. to Yd = 37.3%)

Label 195-130-06 * Taken by D. Huang w/o label 8/1/01 for machining.

\Rightarrow Impregnant Yield (including run-out from both samples) = $(4.64 + 14.52 + 19.76) / 60.08 +$
 $= (38.92 / 93.12) \times 100 = \textcircled{41.8\%}$

Performed and Recorded by: 

Directed by: J. (un)

Read and Understood by:

Date

Date

Date

Subject

Cross-Reference (if any)

Initial Data of Graphitized c/c composites via BP Process (4th Trial) (c/c comp BP) 7
Ref. 145-129-48 (composites at 3000°C graph temp).

Purpose:

To obtain the initial weights and dimensions prior to vacuum impregnation w/ "T-143" type phenolic/Furfural resin blend for densification.

Materials:

c/c composites via BP process. Rec'd from P. Sirotek 8/7/01. Two sections, both graphitized to ~3200°C. Section "A-2" had one pitch impregnation, section "B-2" did not.

Made w/ 0.25" K-23 SE pitch fibers and Reilly 155 pitch. Load ratio = 75/25. No soft fur. Block 13 of 4th Lawrenceburg trial.

Procedure:

Ref. 145-129-96

Initial Data: (8/8/01)

File Path = c:\Program Files\Excel\BP c-c composites\Initial.xls Sheet = BP IV (Appendix)

Material:

Material: BP-IV-13 A2 and BP-IV-13 B2. Rec'd 8/07/01. Ultrasonic washed 1x for 5 min. in deionized water on 8/07/01. Dimensions were obtained with a Mitutoyo Model CD-8'CS digital caliper. Hot vacuum dried at 99 °C to 0.1 mm pressure from 8/07 to 8/08/01. Weights obtained on Mettler PN 2210 balance on 8/08/01.
Note: Both samples have been graphitized to ~3200°C. A2 has one PI. B2 has no PI.

Sample I.D.	Weight (g)	L1 (mm)	L2 (mm)	L3 (mm)	Ave. Length (mm)	W1 (mm)	W2 (mm)	W3 (mm)	Ave. Width (mm)	H1 (mm)	H2 (mm)	H3 (mm)	Ave. Height (mm)	Vol. (cc)	Den. (g/cc)
4-13-A2	266.87	107.91	108.23	108.10	108.08	93.92	93.98	94.11	94.00	15.16	15.29	15.38	15.28	155.209	1.719
4-13-B2	257.29	107.46	107.84	108.12	107.81	97.75	98.11	98.78	98.21	15.34	15.49	15.34	15.39	162.950	1.579

Dimensioned: 08/08/01
Hot Vac. Dried: 08/07-08/01
Weighed: 08/08/01
N.B. Ref. No.: 195-130-07

#

Impregnation

NO REF.

4-13-A2 195-129-53 60/50 vol% GP5432/Furfural

195-130-08-09

4-13-B2 " " " " " " " " " "

195-130-10-11

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

8 Subject VI of 195-130-07#4-13-A2 w/195-129-53 (50/50 vol/10 GP-5432/Kurtom) (BPC com)
 Cross-Reference (if any)

Purpose:

To densify a graphitized c/c composite w/ phenolic / furfural resin blend. Proposed use is a Li cell.

Materials:

- 1) c/c Composite: 195-130-07#4-13-A2 (c/c composite via BP process. From the 13th block of the 4th Lawrenceburg trial. 0.25" long K-223-SE pitch fibers + Kevlar 155 pitch, Load Ratio = 75% w/o sulfur. Picked & rebaked. Graphitized to $\sim 3200^\circ\text{C}$. $W_{\text{wet}} = 246.87\text{g}$, $V_{\text{load}} = 155.209\text{cc}$ Density = 1.719g/cc
- 2) Impregnant: 195-129-53 (50/50 by vol. GP-5432/Kurtom) prep 5/14/01. Stored in refrigerator. Last Used: 7/31/01. $V_{\text{load}} = 17.4\text{cps at } 74.5^\circ\text{F}$, $S.G._{\text{wet}} = 1.188\text{ at } 74.5^\circ\text{F}$. 1. mol m.c.c. = 34.4 ($\sigma = 0.26, n = 3$).

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 & 16. * Processed w/195-130-07#4-13-A2

Pump-down Data: (8/8 & 9/01)

DATE	TIME	PRESS (mtorr)	Comments
8/8	13:00	18	Load core from hot vac. oven (132°C , 0.4 mm)
"	13:10	"	Begin pump-down
"	14:10	35	
"	16:00	25	
8/9	8:15	22	Charge traps w/ dry ice-acetone.
"	8:40	13	LDR
"	9:45	12	Begin V.I.

Impregnation Data: (8/9 & 10/01) - LDR w/ traps charged

LDR: Initial = 13 millitorr $V_{\text{load}} = 17.4\text{cps at } 77.7^\circ\text{F}$ Prep Time = 9:45 (12 m to)
 5 min = 25 " $S.G._{\text{wet}} = 1.192\text{ at } 77.7^\circ\text{F}$ Unload Time = 7:45 8/10/01
 10 min = 34 "
 15 min = 44 "

Comments:

300 ml of impregnant in the 500 ml cylindrical funnel.

cont'd next page.

Performed and Recorded by: *[Signature]*

Directed by: *[Signature]*

Read and Understood by:

Date

Date

Date

Subject

Cross-Reference (if any)

VI of 195-130-07 #4-13-A2 w/ 195-129-53

(c/c comp BP).

9

Post Impregnation Data: (8/10/01)

Wt (PVI-1) = 302.75g \Rightarrow Wt. Pickup = 36.08g, \Rightarrow Wt/lo Pickup = 13.52, Vol/lo Pickup = 19.50

Curing Data: (8/10/01) - Cured w/ vial 195-130-07 #4-13-B2. Samples were placed atop a s.s. screen over a small Al pan. to determine the amount of cured run-out.

Al pan + Screen Wt (g) = 190.95g

	Time	oven Temp	oven Press.	Comments
* (2)	8:05	154	Atm	Load over, Purge w/ argon at 5.0 SCFH (AIR).
- (2)	10:35	150	"	Unload to desiccator. Set over at "82" (50%). Cool composite \Rightarrow weigh.
	-	-	-	Wt = 281.70g \Rightarrow Wt Pickup = 14.83g \Rightarrow Wt/lo Pickup = 5.56 (Yld = 41.1%)
* (2)	12:35	248	Atm	Load over; rotate 180°, reverse position. Purge w/ argon at 5.0 SCFH (AIR).
- (2)	14:31	252	"	Unload to desiccator. Set over "29" (50%). Cool \Rightarrow weigh.
	-	-	-	Wt = 280.12g \Rightarrow Wt Pickup = 13.25g \Rightarrow Wt/lo Pickup = 4.96 (Yld = 36.7%)

Comments:

1) After 2 hrs, ~152°C, atm. pressure;

Set over at "82" (50%). Evidence of run-out on the screen and in Al pan.

Rotate sample 180° (Front of vial). Unload to desiccator \Rightarrow cool \Rightarrow weigh.

2) After 2 hrs, ~250°C, atm. pressure;

Set over at "29" (50%). Unload to desiccator \Rightarrow cool \Rightarrow weigh.

Post Curing Data: (8/10/01)

Pan + Screen + Cured Run-out (g) = 194.79g \Rightarrow Cured run-out = 3.84g

Wt (g) = 280.12g \Rightarrow Wt Pickup = 13.25g \Rightarrow Wt/lo Pickup = 4.96, (Insitu % Yld = 36.7)

Label 195-130-07 #4-13-A2 Given to P. Simocky 8/13/01

Impregnate Yield Including Run-out: Ref. 195-130-11

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject VI of 195-130-07 #4-13-B2 w/195-129-53 (50/50 V₂ 1/6 6P-5432/Referral) (c/c comp BP) 11
 Cross-Reference (if any)

Curing Data: (8/10/01) - Cured w/ VI of 195-130-07 #4-13-B2. Samples were placed atop a s.s. screen over a small Al pan. to determine the amount of cured run-out.

Al pan + screen wt = 190.95g

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:05	154	Atm	Load over. Purge w/argon at 5.0 SCFH (AIR). Oven set at "48" (58%).
-(2)	10:35	150	"	Unload to desiccator. Set oven at "82" (50%). Purge w/argon at 5.0 SCFH (AIR)
	-	-	-	Wt = 278.68g \Rightarrow Wt Pickup = 21.39g \Rightarrow Wt to Pickup = <u>8.31</u> (Yd = <u>42.1%</u>)
*	12:35	248	Atm	Load over; rotate 180°, reverse position. Purge w/argon at 5.0 SCFH (AIR).
-(2)	14:35	252	"	Unload to desiccator. Set oven at "29" (50%). Cool \Rightarrow weigh.
	-	-	-	Wt = 276.35g \Rightarrow Wt Pickup = 19.06g \Rightarrow Wt to Pickup = <u>7.41</u> (Yd = <u>37.5%</u>)

Comments:

1) After 2 1/2 hrs, 152°C, atm. pressure;
 Set oven at "82" (50%). Evidence of run-out on the screen + in the Al pan.
 Rotate sample 180° (rear of oven). Unload to desiccator \Rightarrow cool \Rightarrow weigh.

2) After 2 hrs, 250°C, atm. pressure;
 Set oven at "29" (50%). Unload to desiccator \Rightarrow cool \Rightarrow weigh.

Post Curing Data: (8/10/01)

Pan + Screen + Cured Run-out (cr) = 194.79g \Rightarrow Cured Run-out = 3.84g

Wt (cr) = 276.35g \Rightarrow Wt Pickup = 19.06g \Rightarrow Wt to Pickup = (7.41) (Insitu % Yd = 37.5)

Impregnant Yield (including run-out) = $[(3.84 + 19.06 + 13.25) / (36.08 + 50.85)] \times 100$
 $= (36.15 / 86.93) \times 100 = 41.6\%$

this agrees well w/ impregnant yield from A1 + B1 samples
 Ref. 195-130-06 (i.e. 41.8% yield).

Labeled 195-130-11 #4-13-B2 Given to P. Sirocky 8/13/01

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject Graphitization of 195-129-93 #3-1-B, 195-129-100 #3-1-A, & 195-130-02 #3-2-B (also 63)
 Cross-Reference (if any)

For convenience samples are new labeled 195-130-62 #3-1-B, 3-2-A, & 3-2-B.
 Returned to F. S. Docky 10/01/01.

Material:

195-130-62 #3-1-B: Previously 195-129-93 #3-1-B; c/c composite via RF process from the 1st block of the 3rd Lawrenceburg trial. 0.25" long K-223-SE pitch fibers and Rayley 155 pitch. Load ratio = 75/25. No sulfur. Used w/ silicon oxide colloidal dispersion. $\lambda \rightarrow$ vacuum dried to 160°C to 0.4 mm, wt = 822.45g, Volume = 554.502cc, Density = 1.485g/cc, Wt loss = 863.85g

Graph Cycle:

Held 5 hrs at ~1690°C \rightarrow 1 hr at ~2000°C. Block wrapped in Grafoil sheet.

Graph Run Data:

RUN NO. 2 OF 4

NO. PIECES 1

OPERATOR Harlow m.k.

SIZE _____

N2 _____

①

2

RATE FAST TO 1690 hold 5 hr 3 min TO 2000

ARGON _____

0-2400 SCHED. ACTUAL
 HR. TIME TEMP. TEMP.

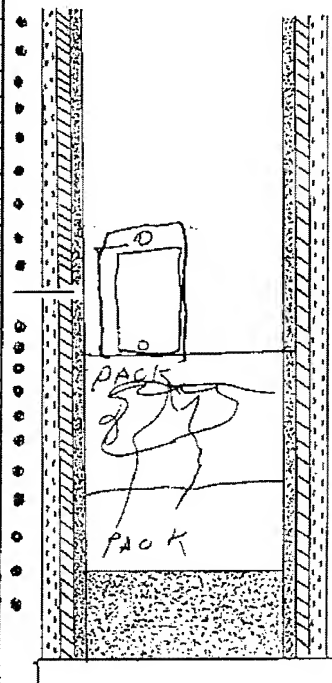
GROUND Hold 1 hr

CURRENT 285 FREQ. 425

HR. TIME	TEMP.	TEMP.	VOLTS	AMPS	% KW
4	R+	18	26	15	7
5		701	41	21	15
6		1225	53	25	23
7		1690	63	33	31
8	1690	1690	46	23	18
9	1690	1695	45	22	17
10	1690	1705	45	22	17
11	1690	1700	45	22	17
12	1690	1700	43	21	16
1230	2000	2000	78	35	35
1330	2000	2010	54	24	22

LOADING DIAGRAM

COMMENTS



925
 SIF TUBE

Performed and Recorded by: _____

Directed by: _____

Read and Understood by: _____

Date _____

Date _____

Date _____

64

Subject

Cross-Reference (if any)

Post Graph Comments:

Very little sic on the sample. Same is true for the grafol wrap.

Material:

195-130-63 #3-2-A: Previously 195-129-100#3-1-A: c/c composite via BP process from 2nd block of the 3rd Lawrenceburg trials. 0.25" long K-223-SE pitch fibers and Re. 71ey 10 pitch. Load Ratio = 25/25. No sulfur. Visc w/ silicate oxide colloidal dispersion 2x = hot vacuum dried after each v.f. Final vac dry (166°C, 0.3mm). Wt (as) = 867.13g. Vol (as) = 516.247g/cc, Density (as) = 1.559g/cc, Wt (PVD-23) = 926.16g

Graph Cycle:

Held 5 hrs at ~1700°C ⇒ 1 hr hold at ~2400°C. Block wrapped in Grafol sheet.

Graph Run Data:

COIL NO. 10DATE 9-20-01SUBMITTER D. HUANCARUN NO. 3 OF 4NO. PIECES 1OPERATOR JIM HARTEN MIKE

SIZE

N2

① 2

RATE 1700° 5 hrs → 2400° 1 hr

ARGON

0-2400 SCHED. ACTUAL
HR. TIME TEMP. TEMP.

VOLTS

AMPS

% KW

GROUND

CURRENT

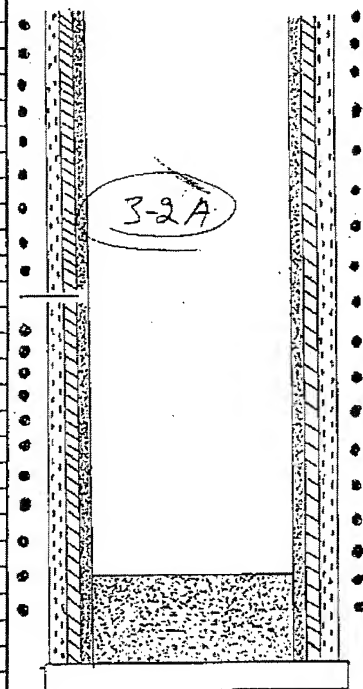
301 FREQ. 428

2	RT	20	27	15	7
3	700	670	40	21	15
4	1200	1210	53	25	23
5	1700	1695	64	32	32
6	1700	1695	47	23	19
7	1700	1690	45	21	17
8	1700	1705	44	21	17
9	1700	1700	43	21	16
10	1700	1705	44	21	17
11	2050	2110	69	35	35
1195	2400	2400	81	47	47
1245	2400	2405	67	34	33

LOADING DIAGRAM

COMMENTS

x785g

Performed and Recorded by: J. HartenDirected by: J. Chen

Read and Understood by:

Date

Date

Date

(SLC Comp BP) 6

Most visible SiC on surfaces of the block of the three samples run here. Appreciably more SiC on the Gratiol Wrap than the block taken to 2000°C.

195-130-63 #3-2-B: Previously 195-130-02 #3-2-A: ck composite via BP process from the 2nd block of the 3rd Lawrenceburg trial. 0.25" long K-223-SE pitch fibers and Reiley 155 pitch. Load Ratio = 70/25. No sulfur. Viedal silican oxide colloidal dispersion 2x \Rightarrow hot vacuum dry after each VT. Final vac dry (466°C, 0.3mm). Wtcs = 861.58g, Volcs = 571.400cc, Dencs = 1.508g/cc, Wt (prev-2) = 929.57g

Held Stns at $\sim 1700^\circ\text{C}$ \Rightarrow 1 hr hold at $\sim 2600^\circ\text{C}$. Block wrapped in Grafoil sheet.

COIL NO. 10

DATE 7-25-01

SUBMITTER D. H. HARRIS

RUN NO. 474

NO. PIECES 1

OPERATOR JIM-HARRIS-MIKE

SIZE

N2

① 2

RATE FAST 1700' HOLD 5 HRS 300'/HR \rightarrow 2600'

ARGON

0 - 2400	SCHED.	ACTUAL
HR. TIME	TEMP.	TEMP.

GROUND *HOLD THR*
CURRENT *290* FREQ. *426*

Performed and Recorded by: *T. Russo*
Directed by: *J. C. Carr*
Read and Understood by:

Date /
Date
Date

Subject *Graph of 195-129-63 #3-1-B, 195-129-100 #3-2-A, + 195-130-02 #3-2-B (c/c comp BP)*
 Cross-Reference (if any)

Post Graph Comments:

Visible SiC on sample's surfaces. Possibly less than 195-130-63 #3-2-A.
 Of three samples this material had the most coating of SiC on the Grafoil
 wrap. The Grafoil wrap was given to J. Norley to evaluate potential of SiC
 coated Grafoil.

Post Graph Data: (9/28/01)

File Path = C:\Program Files\Excel\BP C-C Composites\Post Graph.xls Sheet = BP III

BP C/C COMPOSITES POST GRAPH WEIGHTS AND DIMENSIONS

Material:

Material: BP-III-1 and BP-III-2. Rec'd 09/28/01. Samples not washed (ie. Vac oven out of order).
 Dimensions were obtained with a Starrett No. 123-12 vernier caliper.
 Weights obtained on Mettler PN 2210 balance on 9/28/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-1-B	810.66	8.257	8.277	8.285	8.277	3.259	3.319	3.327	3.305	1.254	1.149	1.228	1.210	542.540	1.494
3-2-A	852.69	8.939	8.938	8.956	8.944	3.245	3.304	3.316	3.288	1.209	1.119	1.195	1.174	565.998	1.507
3-2-B	836.79	8.978	8.982	8.993	8.984	3.295	3.332	3.324	3.317	1.162	1.165	1.165	1.164	568.439	1.472

Dimensioned: 09/28/01

Hot Vac. Dried: N/A

Weighed: 09/28/01

N.B. Ref. No. : 195-130-63

Sample I.D.	Initial through Graph						Post Vacuum Dry through Graph					
	Wt/o	Len/o	W/o	H/o	Vol/o	Dens/o	Wt/o	Len/o	W/o	H/o	Vol/o	Dens/c
3-1-B	-1.58	0.96	1.01	-4.12	-2.16	0.61	-6.15	NM	NM	NM	NM	1.228
3-2-A	-1.67	0.94	0.77	0.00	1.75	-3.34	-7.93	NM	NM	NM	NM	1.195
3-2-B	-2.88	0.89	1.16	-2.51	-0.52	-2.39	-9.98	NM	NM	NM	NM	1.165

Comments:

Dimensionally the sample shrinks in thickness (ie. height). This results
 in expansion in the other two directions.

No where near 1.70 final density desired.

Samples Returned to P. Strackey 10/01/01.

Performed and Recorded by: *[Signature]*

Directed by: *[Signature]*

Read and Understood by:

Date

Date

Date

Subject: Initial Data - C/C Composite via BP Process (1st Trial - no graph) (C/C comp BP) 79
Cross-Reference (if any)

Purpose:

To obtain the initial weights & dimensions prior to vacuum impregnation w/ "T-143" type phenolic/furfural resin blend for densification.

Material:

C/C composite via BP process. Rec'd from P. S. Rocky 10/23/01. This was a slab from the 7th brick of the 1st trial. Slab was not graphitized. Made w/ 0.25" K-223-SE pitch fibers, Reilly NST pitch, and sulfur. The load ratio = 75/25 + 50 wt% of pitch sulfur.

Procedure:

Ultrasonic washed in deionized water 3x, 5 min intervals \Rightarrow Vacuum dried overnight in National Vac. Oven (E-15) overnight at $\sim 98^{\circ}\text{C}$ to 0.5 mm pressure. Cooled to R.T. in desiccator \Rightarrow weighed.

Initial Data: (10/23+24/01)

File Path = C:\Program Files\Excel\BP C-C Composites\Initial.xls Sheet = BP I

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-I-7: not graphitized. Rec'd 10/23/01. Ultrasonic washed 3x for 5 min. in deionized water on 10/23/01. Dimensions were obtained with a Starrett No. 123-12 vernier caliper. Hot vacuum dried at 98°C to 0.5 mm pressure from 10/23 to 10/24/01. Weights obtained on Mettler PN 2210 balance on 10/24/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
1-7-A	429.50	8.060	8.050	8.042	8.051	2.648	2.677	2.703	2.676	0.836	0.795	0.750	0.794	280.192	1.533

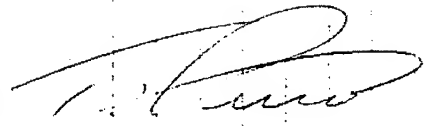
Dimensioned: 10/23/01

Hot Vac. Dried: 10/23-10/24/01

Weighed: 10/24/01

N.B. Ref. No. : 195-130-79

1st VI w/ T-143: Ref. 195-130-81+82

Performed and Recorded by: 
Directed by: J.C. Curran
Read and Understood by:

Date
Date
Date

80

Subject Preparation of ~80 ml of sol'n by Vol GP-5432 from 101
 Cross-Reference (if any) Previous treat: Ref 195-129-53

(calc comp Br

Purpose:

To ensure enough impregnant for 2 VIs of 195-130-79 #1-7A. This will be combined w/ 195-129-53 prior to impregnation.

Materials:

GP-5432, Lot # 19588. Rec'd from Georgia-Pacific 9/24/96, LIMS # A96-036315.
 Stored in freezer in E-13, Ave % Mac MCC = 48.6 ($50-6.37, n=3$), Brookfield
 RT Viscosity = 157.3 cP at 76.0°F , PDSC Data: 2 $^{\text{nd}}$ 195-105-49, TGA Yield
 (900°C) = 47.5% , Brookfield Visc (5/14/01) = 283.5 cP at 68.7°F .

Furfural: Reagent Grade (Fisher), Rec'd 5/15/01. 12" bottle. Use ~250 ml
 \Rightarrow Balance = ~640 ml.

Preparation: (10/25/01) - 1L Erlenmeyer Flask. 250 ml GP-5432 + 250 ml Furfural
 Stir for ~30 min, & topped off \Rightarrow combine w/ 195-129-53
 Obtain Brookfield (LVT) RT viscosity and RT Specific Gravity

FLASK (etc) + GP-5432 wt = 734.0	FLASK (etc) + Furfural wt = 1020.3
FLASK (etc) wt = 430.8	FLASK (etc) wt = 734.0
~250 ml GP-5432 wt = 303.2 g	~250 ml Furfural wt = 286.3 g \Rightarrow 51.4% by wt GP-5432

Mix w/o heat for ~30 min \Rightarrow combine w/ 195-129-53


Brookfield (LVT) RT Viscosity: (10/25/01)

Viscosity = 19.9 cP at 74.2°F Spindle #1, CORPM, Factor = 1

Specific Gravity at RT: (10/25/01)

S.G. = 1.194 at 74.2°F

Label combined solutions 195-130-80. Store in refrigerator when not in use.

Performed and Recorded by: 
 Directed by: J. Chini
 Read and Understood by:

Date
 Date
 Date

Subject 1st VI of 195-130-79 #1-7-A w/195-130-80 (sol/sol by vol GP-5432/FurFural) (c/c comp BP) 81
Cross-Reference (if any)

Purpose:

To density c/c composite produced via BP process. Note: this composite has not been graphitized.

Materials:

- 1) c/c Composite: 195-130-79 #1-7-A (c/c composite via BP process) Rec'd From P. Smocky 10/23/01
 (7th block of 1st Lawrenceburg Trial, not graphitized), 0.25" long K-223-SE pitch fibers
 : Reilley 105 pitch + sulfur. Load ratio = 75/25 + 5 gp H wt/o of pitch = 50 Hwt
 Wt/Lt = 429.50 g, Vol (cc) = 280.192 cc, Density = 1.533 g/cc
- 2) Impregnant: 195-130-80 (sol/sol by volume GP-5432/FurFural). Prep. 5/14/01. Refrigerated.
 Add'l sol'n (Y/N): (195-130-80) and combined. Last Used: 8/10/01. New Initial Data:
 Visc (cP) = 19.9 cP at 74.2°F, S.G. (wt) = 1.194 at 74.2°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16

Pump-down Data: (10/24+25/01)

DATE	TIME	PRESS (mTorr)	Comments
10/24	13:25	9	Load core from desiccator.
"	13:35	"	Begin pump-down
"	14:45	26	
"	16:00	18	
10/25	7:25	12	
"	9:05	13	Charge traps w/dry ice-acetone
"	9:35	7	LDR
"	11:45	6	Begin VI

Impregnation Data: (10/25+26/01) - LDR w/traps charged

LDR: Initial = 7 mTorr Visc (cP) = 19.9 cP at 74.2°F Drop Time = 11:45 (6 mTorr)
 5min = 16 " S.G. (wt) = 1.194 at 74.2°F Unload Time = 8:45 (10/26/01)
 10min = 23 " Held at atmospheric pressure
 15min = 30 " for ~ 21 hrs.

Comments:

500 ml cylindrical Funnel is full (ie ~640 cc). ~30 ml returned to 16 oz bottle.
 ⇒ plenty for at least 3 more VIs.

cont'd next page

Performed and Recorded by:

Directed by: J (lw)

Read and Understood by:

Date,

Date

Date

Post Integration Data: (10/26/01)

Wt (PVI-1) = 494.21g \Rightarrow Wt. Pickup = 61.81g \Rightarrow Wt. Pickup = 15.09, Vol% Pickup = 19.61
 Al Fuel + Sample Wt = 196.94g

Curing Data: (10/26/01) - Placed atop s.s. screen at small Al pan. Held ~2 hrs at ~100°C \Rightarrow ~2 hrs at ~200°C, purged at 20 SCFH (AIR) w/42.5. Throughout.
 First Use of Fisher Scientific Iso-temp Vacuum Oven (Model #2E2A).

	Time	Temp (Temp)	Press (mm)	Comments
+	4:00	153	Atm	Load oven. Over set at 153°C. Purge nitrogen at 20 SCFH (AIR)
- (2)	11:00	153	"	Unload in desiccator. Set oven at 200°C
-	-	-	-	Wt = 462.86g \Rightarrow Wt. Pickup = 33.36g \Rightarrow Wt. Pickup = 7.77 (Vol% = 51.5%)
+	12:00	275	Atm	Load oven, rotate 180°. Set oven at 200°C. Purge nitrogen at 20 SCFH (AIR)
- (2)	14:00	271	"	Unload in desiccator. Set oven at 100°C. Allow sample to cool through
-	-	-	-	Wt = 457.84g \Rightarrow Wt. Pickup = 28.34g \Rightarrow Wt. Pickup = 6.66 (Vol% = 43.3%)

Comments:

1) After 2 hrs, ~153°C, at pressure;
 Set oven at 200°C. Screen 4 pan contains minor amount of cured resin \Rightarrow run-out. Top surface has small "bubbles", while bottom surface has more "droplets". Rotate 180° (i.e. droplets to top).

1) After 2 hrs, ~275°C, at pressure;
 Set oven to 200°C. Unload in desiccator \Rightarrow cool \Rightarrow weigh. Sample "bubbles" "droplets" from composite surface \Rightarrow reweigh. Store in desiccator over weekend. Load into oven at 100°C at 10/29/01.

Post Cure Data:

Sample weighed \Rightarrow reweighed.

Wt = 497.58g \Rightarrow Wt. Pickup = 28.08g \Rightarrow Wt. Pickup = 6.81, Vol% = 43.3%

Resin + cured Resin (Wt) = 196.28g \Rightarrow Cured content = 0.24g

Total Cured Resin (includes cured resin scraped from composite) = 0.24 + 0.24g = 0.48g

Impregnated Field (including run-out + scrapings) = [60.66g + 28.08g] / 64.81g \times 100 = 44.3%

2nd VI: Ref. 195-130-85+86

Performed and Recorded by: [Signature]

Directed by: J. C. [Signature]

Read and Understood by: [Signature]

Date: [Signature]
 Date: [Signature]
 Date: [Signature]

Subject 2nd VT of 195-130-82#1-7-A w/195-130-80 (50100 vol/10 GP-5432 / Futrol) (c/c comp BP) 85
 Cross-Reference (if any)

Purpose:

Ref. 195-130-81

Materials:

- 1) c/c composite via BP process: 195-130-82#1-7-A, Rec'd from P. Sirocky 10/23/01. (7th Block of 1st Lawrenceburg Trial; not graphitized). 0.25" long K-223-SE pitch fibers, Reiley 155 pitch, sulfur, Load Ratio = 72/25 + 5ppH wt% of pitch = sulfur. Vied and cured 1x w/195-130-80. Wt(15) = 429.50g, Vol(15) = 280.192cc, Dens(15) = 1.533g/cc, Wt(PVP-1) = 457.58g, Current Den = 1.633g/cc
- 2) Impregnant: 195-130-80 (50100 by volume GP-5432 / Futrol) Prep. 5/14/01 and 10/25/01. Last Used: 10/20/01. Visc(15) = 19.9 cps at 74.2°F, S.G. (15) = 1.194 at 74.2°F. Refrigerated when not in use.

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15+16

Pump-down Data: (10/29+30/01)

DATE	TIME	PRESS (mTorr)	Comments
10/29	8:30	9	Load core from warm over; ~94°C, atm. pressure.
"	8:40	"	Begin pump-down
"	9:40	105	
"	10:00	46	
10/30	7:30	26	
"	8:10	27	Charge traps w/ dry ice-acetone
"	9:15	18	LDR
"	11:45	18	Begin VI

Impregnation Data: (10/30+31/01) - LDR w/4 traps charged.

LDR: Initial = 18 mTorr Visc(15) = 18.7 cps at 76.9°F

5min = 35 " S.G. (15) = 1.193 at 76.9°F

10min = 47 "

15min = 53 "

Drop Time = 11:45 (18 mTorr)

Unload Time = 8:45 (10/31/01)

Held at atmospheric pressure for

~21 hrs.

Comments:

~610ml in 500cc. cylindrical Funnel → Enough for another impregnation.

Post Impregnation Data: (10/31/01)

Wt(PVP-2) = 491.45g ⇒ Wt. Pickup = 33.87g ⇒ Wt to Pickup = 7.40, Vol to Pickup = 10.13

Performed and Recorded by: [Signature]

Directed by: [Signature]

Read and Understood by:

Date

Date

Date

Subject 2^{nd} VI of 195-130-82 #1-7-A w/195-130-86 (50/50 vol% GR 532 / Air Dry) (1k comp BP)
 Cross-Reference (if any)

Curing Data: (10/31/01).

Procedure: Ref. 195-130-82

AI Pan + Screen wt = 190.97g

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:55	156	Atm	Load oven. Oven set <u>155°C</u> . Pan w/always at 50.300 (Atm).
(2)	10:55	154	"	Unload to desiccator. Set oven at <u>265°C</u> .
	-	-	-	Wt = 480.63g \Rightarrow Wt Pickup = 23.25g \Rightarrow Wt/c Pickup = 5.08 (Yld = <u>68.6%</u>)
*	12:00	275	Atm	Load oven, rotate 180°. Set oven at <u>260°C</u> , Pan w/always at 50.300 (Atm)
(2)	14:00	271	"	Unload to desiccator. Set oven at <u>105°C</u> . Allow sample to cool \Rightarrow weigh \Rightarrow set
	-	-	-	Wt = 474.25g \Rightarrow Wt Pickup = 18.71g \Rightarrow Wt/c Pickup = 4.09 (Yld = <u>58.2%</u>)

Notes:

1) After 2hrs, 155°C, atm pressure;

Set oven at 265°C. Transfer to desiccator \Rightarrow cool \Rightarrow weigh. Composite has numerous cured resin bubbles on all surfaces. Rotate 180°.

2) After 2hrs, 273°C, atm. pressure;

Set oven at 105°C. Transfer to desiccator. Cool \Rightarrow weigh \Rightarrow scrape "bubbles" \Rightarrow reweigh. Store in desiccator.

Post Cure Data: (10/31/01)

Sample scraped \Rightarrow reweighed!

Final Wt = 474.63g \Rightarrow Wt. Pickup = 17.05g \Rightarrow Wt/c Pickup = 3.73 Yield = 58.3%

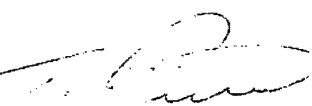
Pan + Screen (Cured) Run-out = 191.18g \Rightarrow Cured Run-out = 0.31g

Total Cured Run-out (includes cured resin scraped from composite) = 0.21 + 0.66 = 1.87

Ingrain Yield (includes run-out scraping) = $[(1.87 + 17.05) / 33.87] \times 100 = \underline{55.9\%}$

Cumulative Wt Pickup = 45.13g \Rightarrow Cumulative Wt/c Pickup = 10.51

Label in plastic bag: 195-130-86 # 1-7-A.

Performed and Recorded by: 

Directed by: 

Read and Understood by:

Date

Date

Date